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AFRPL-TR-66-123

(U) SOLID PROPELLANT EXPLORATORY EVALUATION SEMIANNUAL REPORT NO. 3

376083

F. W. VILLAESCUSA, CAPT, USAF

J. E. VINT, LT, USAF

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TECHNICAL REPORT NO. AFRPL-TR-66-123

JUNE 1966

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SOLID PROPELLANT EXPLORATORY EVALUATION
SEMIANNUAL REPORT NO. 3

F. W. Villaescusa, Capt, USAF
J. E. Vint, Lt, USAF
P. H. Nicks, Lt, USAF

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AIR FORCE ROCKET PROPULSION LABORATORY
RESEARCH AND TECHNOLOGY DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
EDWARDS, CALIFORNIA

FOREWORD

This report summarizes the progress on Project 314804012, Solid Propellant Exploratory Evaluation, by the Exploratory Evaluation Branch in the Propellant Division of the Air Force Rocket Propulsion Laboratory from 1 July 1965 to 30 December 1965.

This report has been reviewed and approved.

ELWOOD M. DOUTHETT
Colonel, USAF
Commander, Air Force Rocket Propulsion Laboratory

UNCLASSIFIED ABSTRACT

This report describes the development of a capability to process composite propellants, the evaluation of thermally stable samples of LMH-1, and the work concerned with the desensitization of JNFO-635.

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PART I

DEVELOPMENT OF A COMPOSITE PROPELLANT CAPABILITY

Lt James E. Vint

PART I

DEVELOPMENT OF A COMPOSITE PROPELLANT CAPABILITY

I. ABSTRACT

(U) Composite propellants with high solids loadings were made to gain an in-house capability in processing highly viscous composite propellants.

(U) Viscosities as high as 70 kilopoise were encountered with solids loadings at 86%. Batch sizes varied from 15 grams to 4 pounds. Burn rate, propellant density, and Shore A hardness were determined for the formulations processed.

II. INTRODUCTION

(U) This project began on 1 July 65, and propellants have been formulated using carboxy functional polybutadiene, polyester, and polyurethane binders, with solids loadings up to 86%.

(U) The initial phase involved development of an in-house capability for processing composite solid propellants with viscosities greater than 20 kilopoise.

III. DISCUSSION

A. Background

(U) Previous propellant processing at the AFRPL had involved only double-base systems. The need for evaluating new propellant ingredients in composite binders has become acute.

(U) Processing of composite propellants requires an entirely different technology in that the ingredients must be heated during mixing, and the high viscosities of the propellants eliminate the possibility of casting motors in the manner used for double-base propellants.

(U) Since the principal objective of this program was to develop a capability in composite propellants, the decision was made to first duplicate systems about which there was information available. In this way, a determination could be made as to whether or not the propellant produced had properties similar to the known system.

B. Experimental Techniques and Apparatus

(U) Processing of high-viscosity propellant presented certain basic problems. Ordinary laboratory mixes were found unsatisfactory since a laboratory mixing apparatus, consisting of a stirrer blade in a round-bottom flask, satisfactory for low-viscosity mixtures, pushed the propellant in front of the blade leaving it only partially mixed.

(U) A 1-pint Baker-Perkins vertical mixer with planetary action blades was set up in the AFRPL Formulation Laboratory so that various formulations could be made, using dummy oxidizer (KCl), to gain information on mixing procedures and conditions.

(U) For live mixes a 1-pint Baker-Perkins mixer, which was fitted for remote operations, was used to process propellant for strands and physical properties. Later a 150-cc Atlantic Research Corporation cone vertical mixer was found to be satisfactory for mixing high-viscosity propellant (up to 70 kilopoise mixes have been made). Batches from 15 to 125 grams have been made in this mixer.

(U) For larger mixes to cast motors, a 1-gallon Baker-Perkins vertical mixer has been modified to cast high-viscosity propellant.

(C) The first propellant to be duplicated was TP-H-1001 (1st stage Minuteman I) which was 86% solid material. Composition of this propellant is given in Table I.

(C) Next, TP-H-8038A (2nd stage Blue Scout) was attempted, however, the prepolymer, which was over two years old, had deteriorated to the point that cure could not be effected. Composition of this propellant is given in Table I.

(U) A polyurethane system, CPU-101, was formulated using Estane, a product of the B. F. Goodrich Company, and containing 85% solids. Composition is listed in Table I.

(U) A polyester system using HX-735, a Minnesota Mining and Manufacturing Company polymer, was formulated duplicating United Technology Corporation's PEP-150 binder. This formulation is listed in Table I.

(C) TABLE I

PROPELLANT FORMULATIONS

	TP-H-1001	TP-H-8038A	CPU-101	PEP-150
Aluminum	16.0	14.00	25.0	16.0
Ammonium perchlorate	70.0	70.00	60.0	68.0
PBAN[a]	11.2			
PBAA[b]		13.20		
Estane[c]			14.32	
HX735[d]				6.72
Curing Agent	2.8[e]	2.8[e]	0.86[f]	1.28[g]
Trimethylolethane Trinitrate				8.00
cure temp/time	140°F/96hrs	140°F/96hrs	140°F/30hrs	140°F/48hrs
Mix Temp	145°F	145°F	145°F	145°F

a. Carboxy functional polybutadiene manufactured by American Synthetic Rubber Corp

b. Carboxy functional polybatadiene manufactured by Thiokol Chemical Corp.

c. Polyurethane manufactured by the B. F. Goodrich Company

d. Polyester manufactured by Minnesota Mining and Manufacturing

e. ERL-2795, Union Carbide Corp

f. Trimethylolpropane, Triethanolamine, 1, 4 Butanediol

g. MAPO (Interchemical Corp), Epon 812 (Shell Chemical Co)

(U) Burn rate data was obtained using an Atlantic Research Corporation model 202 strand burner, with 3-inch-long propellant strands, at pressures up to 1300 psi.

(U) Viscosities of uncured propellant were obtained using a Brookfield model HBT viscometer. This instrument has a range of 0 to 1600 kilopoise when used with a Helipath stand.

(U) A vacuum casting and weigh can has been fabricated from Rohm and Haas specifications. This device will allow casting, under vacuum, of highly viscous propellants.

IV. RESULTS AND INTERPRETATION

(U) Shore hardness and densities of TP-H-1001, CPU-101, and PEP-150 are given in Table II.

(U) Burn rates of these propellants are listed in Figure 1.

(U) These results are comparable to published results of the TP-H-1001 and PEP-150 systems. The CPU-101 formulation was devised at the AFRPL and is not a duplicated propellant.

(C) TABLE II

SHORE HARDNESS AND DENSITY OF PROPELLANTS

Formulation	Shore Hardness	Density (25°C)
TP-H-1001	65-75	1.77
CPU-101	80-85	1.94
PEP-150	75-85	1.90

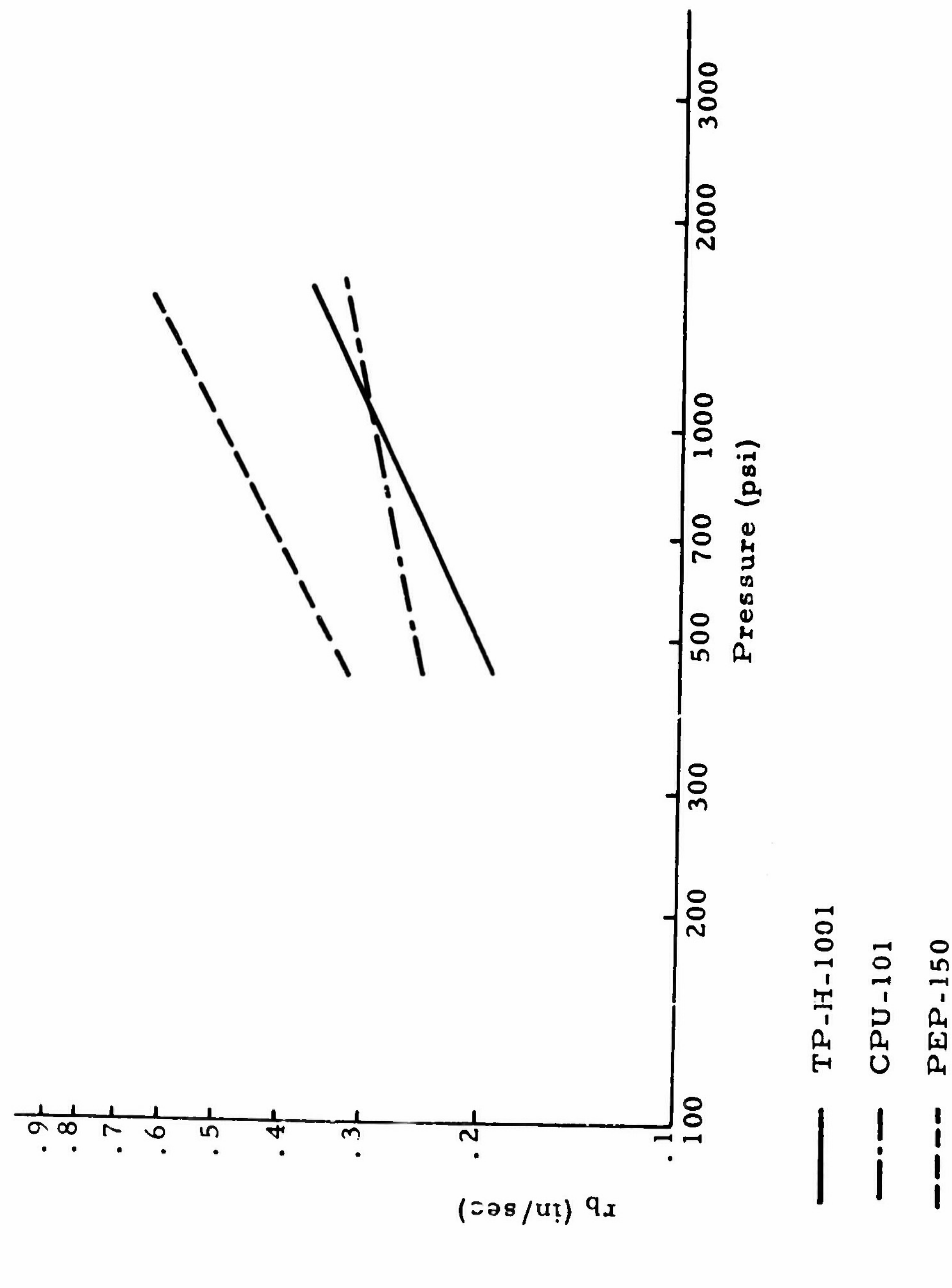

Figure 1. Propellant Burn Rates

V. FUTURE PLANS

(U) One-fourth-lb and 6-lb motors will be cast during the next 2 months, and procedures will be developed for casting viscous propellants using the apparatus previously described.

(U) Casting and mandrel insertion hardware is currently being fabricated by Rohm and Haas Company, Redstone Arsenal Research Division, Huntsville, Alabama.

(U) As soon as a capability for mixing and casting of composite propellant has been established, work will begin on evaluation of the Workhorse Binder currently under development by Aerojet-General Corporation.

PART II

EVALUATION OF THERMALLY STABLE LMH-1 SAMPLES

Capt F. Warren Villaescusa

PART II

EVALUATION OF THERMALLY STABLE LMH-1 SAMPLES

I. ABSTRACT

(C) Thermal-stability determinations were made on three samples of aluminum hydride produced by Olin Mathieson during attempts to prepare a more stable AlH_3. The best sample underwent 1% decomposition in 660 hours at 60°C. Double-base propellant samples were formulated. The most stable AlH_3 yielded the most stable propellant even though the propellant density upon curing was the lowest of the three propellant samples.

II. INTRODUCTION

(C) Olin Mathieson Chemical Corp under contract AF 04(611)-10548 is attempting to prepare a more thermally stable form of AlH_3. Three of the better samples were received and evaluated with respect to thermal stability and formulatability.

III. DISCUSSION

A. Background

(C) Currently, AlH_3 produced in pilot plant quantities is a material that is about 100μ mean particle diameter in size and at 60°C will undergo 1% decomposition in about 5 to 8 days. The material can be washed with acrylonitrile and in this way be made compatible with standard double-base propellant ingredients This treatment has an unpredictable effect on thermal stability, sometimes improving and other times degrading stability.

(C) The three samples of AlH_3 received from Olin had much improved thermal stability. Sample S-288 made by Olin's solid lithium

aluminum hydride process (1) underwent 1% decomposition at 60°C in about 500 hours in Taliani tests at Olin. Two other samples, S-297 and S-298, were from a batch of older material that had been made by Olin's standard solvent process. The samples had been treated with water vapor and an improvement in thermal stability noted. S-297 had been treated in a 5-g batch and S-298 treated in a 20-g batch to determine if there were any batch size effects.

B. Experimental Techniques and Apparatus

(C) A syringe apparatus was used for the gassing determinations. About 0.25g of AlH_3 was placed in a small test tube and the test tube sealed to a 5-cc syringe with a 1-inch piece of Tygon tubing. The apparatus was then placed in an oil bath up to the top of the syringe cylinder.

(U) For propellant gassing, about 1.5 g of freshly mixed propellant was placed in the above apparatus. Propellant used was DB-20.

(C) DB-20 Formulation

Ingredient	Weight Percent
AlH_3	20
AP	30
TMETN	28
TEGDN	9
Nitrocellulose	12
Resorcinol	1

Six gram batches were made. A visual check was made on viscosity and propellant density was checked after curing 20 hours at 40°C.

(C) Particle size determinations were made with a Sharples Micromerograph. All other operations with AlH_3 were conducted in the dry boxes described in Reference 2.

IV. RESULTS AND INTERPRETATION

(C) Figure 1 contains the thermal stability results for the three new AlH_3 samples plus one sample, S-281, received one year ago. S-288 is the best sample, exhibiting 1.0% decomposition after 660 hours. Sample S-297, the smallest water treatment sample, exhibits better stability than S-298. Apparently, care must be taken with the water vapor treatment because, in this instance, there is a batch size effect. The 1-year-old S-281 sample was run just for comparison. However, the aging effect noted by both Dow and Olin is apparent in curves 5 and 6.

(U) When S-288 was formulated in DB-20, propellant density was 93% of theoretical. A sample was acrylonitrile-washed (method described in Reference 3) before formulation in an attempt to improve the propellant density. The treatment was ineffective and propellant density remained at 93%. Thermal stability of S-288 was slightly degraded by the treatment.

(U) Batches of DB-20 with both S-297 and S-298 had densities of 99%. American Potash's regular grade of ammonium perchlorate (AP) was used in all mixes. Attempts to use finer AP resulted in noncastable mixes. The S-297 and S-298 mixes were slightly more fluid than the S-288 mix.

(U) Micromerograph determinations placed the median particle size at 18μ for S-288 and 21μ for S-298. In addition, 21% of S-288 was below 10μ as opposed to only 13% for S-298.

(C) Sample S-298 yielded more dense propellant because of better compatibility with the propellant ingredients. However, as shown in Figure 2, stability of the propellant is much worse than that of the propellant made with S-288. As might be expected the more thermally stable AlH_3 yielded the most stable propellant.

V. FUTURE PLANS

(U) Evaluation of any samples produced by Olin Mathieson will continue.

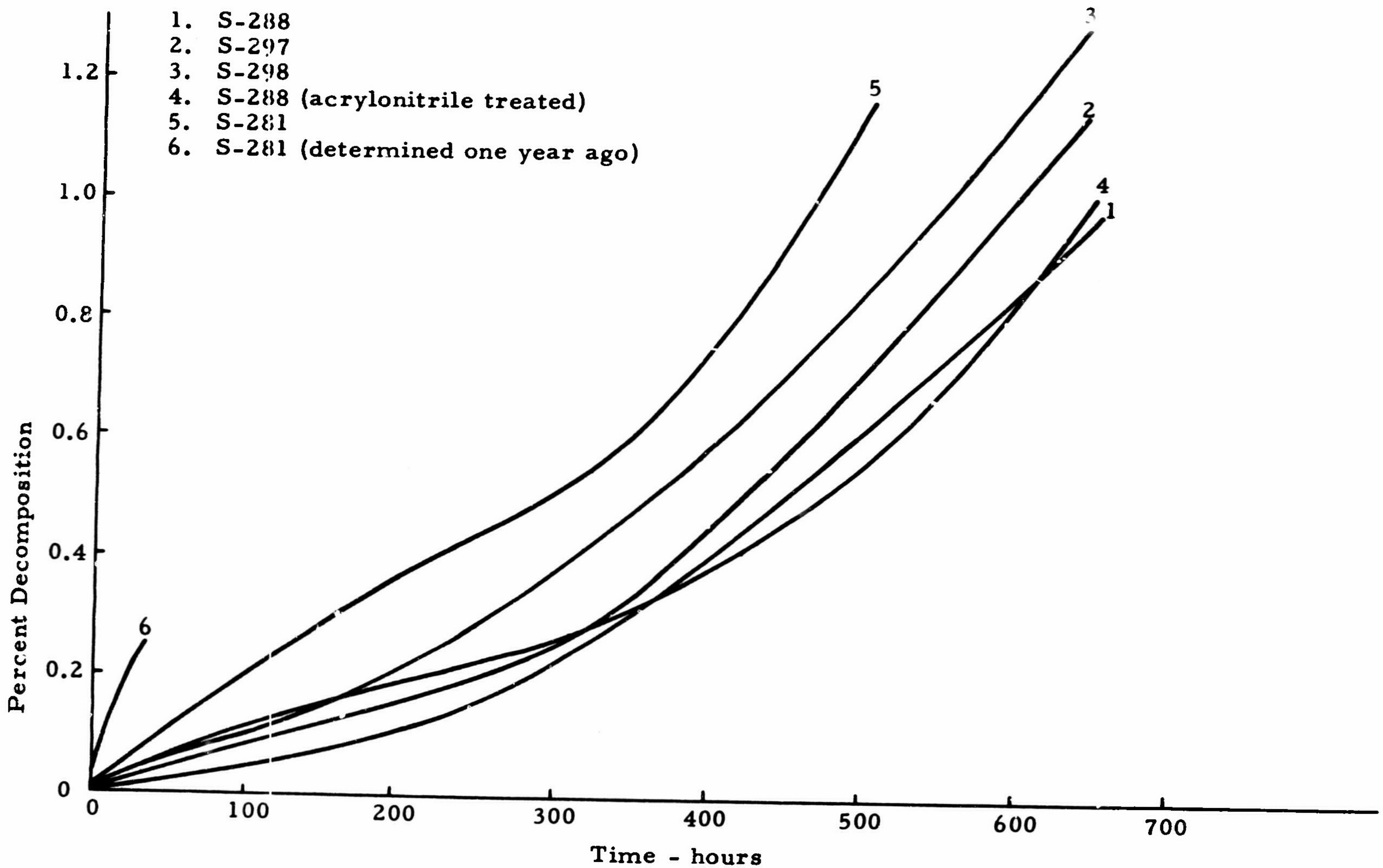

Figure 1. Thermal Stability of Olane 58 at 60°C

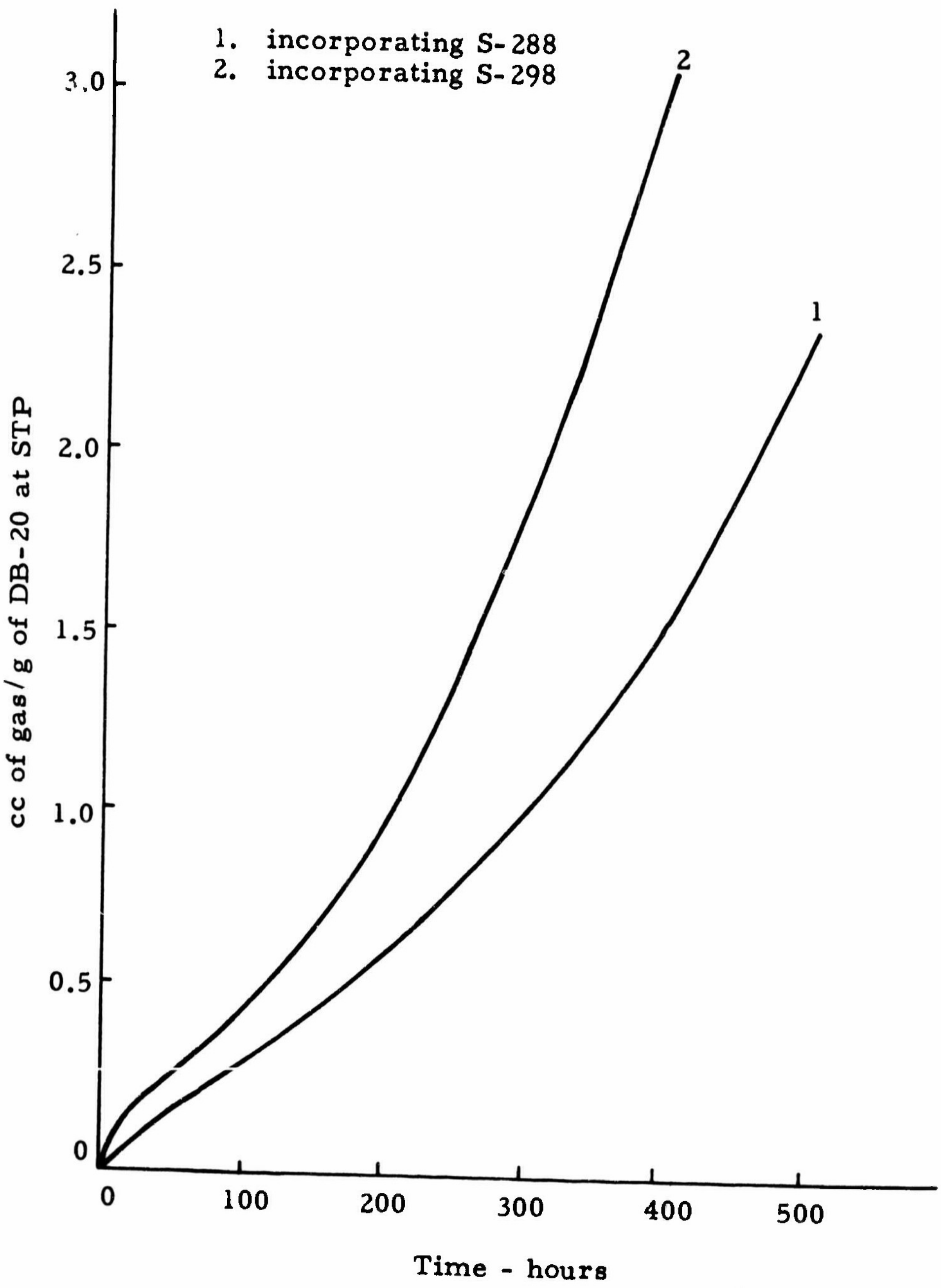

Figure 2. Thermal Stability of DB-20 at 60°C

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1. High Energy Propellant Ingredient Research, Olin Mathieson Chemical Corp, AFRPL-TR-65-132, 30 June 65 (Confidential).

2. Experimental Evaluation of Advanced Propellants, Progress Summary Report Number 3, Air Force Rocket Propulsion Laboratory, RPL-TDR-64-62, May 1964 (Confidential).

3. Solid Propellant Synthesis and Evaluation Semiannual Progress Report No 1, Air Force Rocket Propulsion Laboratory, AFRPL-TR-64-181, December 1964 (Confidential).

PART III

INFO-635 CHARACTERIZATION

Lt Paul H. Nicks
A1C Willard Wooten

PART III

INFO-635 CHARACTERIZATION

I. ABSTRACT

(C) This report describes work concerned with the desensitization of INFO-635, $(NF_2)_3COCH_2CH_2NH_3^+HClO_4^-$(1). No improvements in friction sensitivity were observed when samples of INFO-635 were washed with Freon 11 or 113, which is in direct contrast to the improvement in impact sensitivity noted by this laboratory.

(C) A solid material (probably compound 535, HFNC $(NF_2)_2OCH_2CH_2NH_3^+ClO_4^-$) has been isolated, and samples of this material have been subjected to impact and friction tests. Although sample purity is unknown and may play a major role, preliminary data indicate that this material is quite insensitive to impact and friction.

(U) Differential thermal analysis of a variety of samples of INFO-635 indicated that some ultrasensitive ingredients may have been removed from INFO-635 by the Freon treatments.

II. INTRODUCTION

(U) High-impact, electrostatic and friction sensitivities of energetic NF solid compounds have deterred their potential utilization in solid propellants and hindered NF propellant evaluation. Previous work (2, 3, 4) has been concerned with the preparation, purification and compatibility of INFO-635 with double-base propellant ingredients. The objective of this program is to desensitize these compounds with a minimum resultant loss of energy.

III. DISCUSSION

A. Background

(U) In previous work (5), it was noted that maximum desensitization to impact was achieved by washing INFO-635 with Freon-113 for 5 minutes. Shorter and longer washes proved less effective. A possible explanation for the shape of the curve in Figure 1 of Reference 5 is that some minor impurity is removed, and after 5 to 6 minutes INFO-635 starts to decompose into more sensitive components. To test this hypothesis, INFO-635 was tested for impact and friction sensitivities after extended Freon 11 and 113 washings. Differential thermal analysis (DTA) was performed on a variety of INFO-635 samples to compare the thermal behavior of Freon-treated samples with the non-Freon-treated samples.

(U) Since Freon treatments appeared to improve the impact sensitivity of INFO-635, samples of this Freon-treated material were tested to determine if the fluorocarbon washings had any effect on friction sensitivity.

(C) Compound 535, HFNC $(NF_2)_2$ $OCH_2CH_2NH_3^+HClO_4^-$ differs in structure from INFO-635 by one hydrogen atom. Attempts were made to synthesize Compound 535 to compare its friction and impact sensitivities with that of INFO-635 and with other NF solid compounds.

B. Experimental Techniques and Apparatus

(U) The techniques for treating the INFO-635 samples with Freon are described in Reference 5.

(U) A new friction tester obtained from Esso Research and Engineering Company has been installed at the AFRPL. A small supply of abrasive grits, ranging in Moh hardness from 2 (KCl) through 10 (diamond), was also received with the tester (Table I). The apparatus and its operation is described in Reference 6.

(U) DTA data were obtained with a DuPont 900 Differential Thermal Analyzer and impact data with an Olin Mathieson drop weight tester with a 1-kg weight.

IV. RESULTS AND INTERPRETATIONS

(U) The results obtained with the Screw Friction Tester are given in Table II. Washing INFO-635 with Freon 11 and 113 for periods of 5 minutes, 60 minutes, and 5 hours produced no discernible difference in friction sensitivity. More studies in this area will have to be conducted before a conclusion can be made as to the maximum effect of Freon washings on the friction sensitivity of INFO-635, or to determine if any such relationship exists. INFO-635 washed with Freon-113 for extended time periods (1,5 hours) appeared slightly more sensitive to impact than the original material washed 20 minutes. This is not surprising since our observations have shown INFO-635 to decompose slightly when left in contact with Freon-113 for periods longer than 30 minutes.

(U) Other methods, such as aqueous extraction and liquid chromatography on silica gel, did not improve the friction sensitivity of INFO-635. No relationship between friction sensitivity and purity was observed after purifying the crude material in the above manner. Repeated chromatography of INFO-635 gave a 4°C rise in melting point over the once-chromatographed material, but the impact and friction sensitivities were unchanged.

(C) Small yields of a solid product were obtained from the reaction of perfluoroguanidine (PFG) with ethanolamine perchlorate (EAP). Preliminary data suggest that this material is insensitive to friction and impact. The material also appeared to be highly hygroscopic which can probably be attributed to the presence of some unreacted ethanolamine perchlorate. No attempt was made to purify or specifically identify the product as Compound 535, therefore, no comparison of the sensitivity of this compound to any other NF compound is being made. Its thermal decomposition is quite rapid since the material exploded when heated 3°C beyond its melting point. Results from sensitivity tests conducted on the crude solid product are given in Table II.

(U) Differential thermograms of INFO-635 washed with Freon 11 and 113 showed no endotherm which is in marked contrast to other samples that were not treated with Freon. The untreated samples showed an endotherm at 167°C and a decomposition exotherm at 230°C (Figures 1, 3, 5, 7). The Freon-treated samples showed only an exotherm at 225°C (Figures 2, 4). These curves were reproduced when the heating rate was varied (10°C/min, 30°C/min).

(U) Three samples of INFO-635 were sent to Esso Research and Engineering Co. for analysis (one untreated sample and two treated with Freon-113 for 5 minutes and 60 minutes respectively). Esso's DTA graphs failed to show any significant difference among the three curves (7). All three showed the endotherm at 167°C. This cannot be explained at present, however, there is a possibility that the INFO-635 sent to Esso differed somewhat in sample purity, since samples were obtained from a different batch.

V. FUTURE PLANS

(U) Work in this area will be curtailed due to reassignment of personnel previously working on this project.

TABLE I

ABRASIVE GRIT AND MOHS HARDNESS

MOH HARDNESS	MATERIAL
2	KCl
3	Calcite
4	Fluorspar
5.5	Glass (Pyrex)
6	Agate
7	Quartz
8	Beryl
9	SiC
10	Diamond

TABLE II

FRICTION AND IMPACT TESTS ON INFO-635 AND COMPOUND 535

Sample	Treatment	Melting Point °C	Impact kg - cm	Friction Screw Bare	Friction Screw Fluorspar*
INFO-635	None (Crude)	204	2.5-3.0	0	+
↓	Crude, water-washed	207	7.0	0	+
↓	Crude, Freon-11 washed	204-205	8.5	0	+
↓	Crude, Freon-113 washed (6 minutes)	205	9.5-10.0	0	+
↓	Once chromatographed	209	8.5	0	+
↓	Twice chromatographed	211-212	8.5	0	+
↓	Washed with Freon-113 (60 minutes)	---	6.0	0	+
↓	Washed with Freon-113 (300 minutes)	---	4.0-5.5	0	+
535	None	182**	>15	0	0***

NOTES:

*Fluorspar is only material shown because it is minimum abrasive grit required for explosion

**Exploded when heated to 185°C

***No fire using Diamond, hardest grit available.

+ = fire

0 = no fire

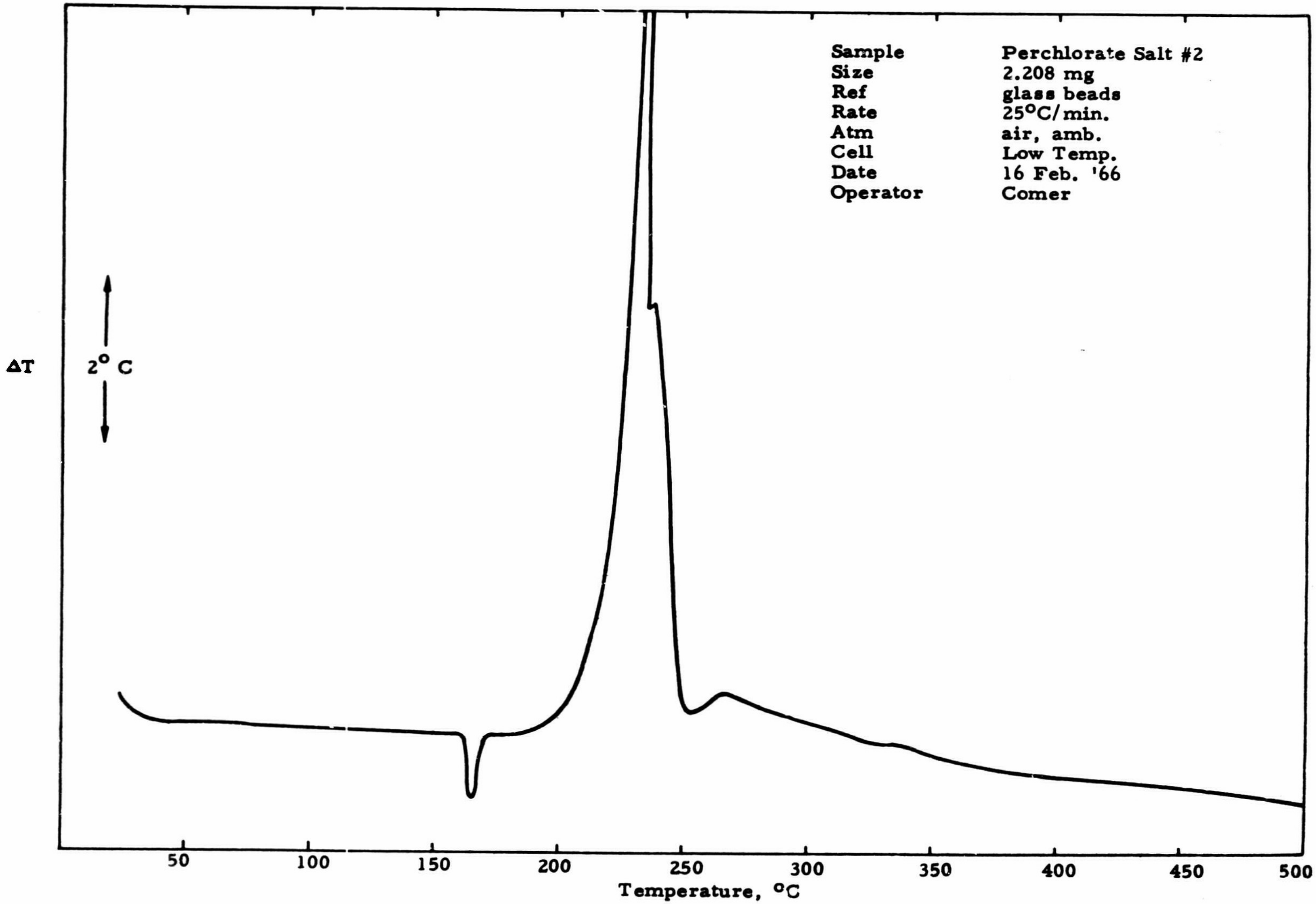

Figure 1. INFO-635 (As received from 3M)

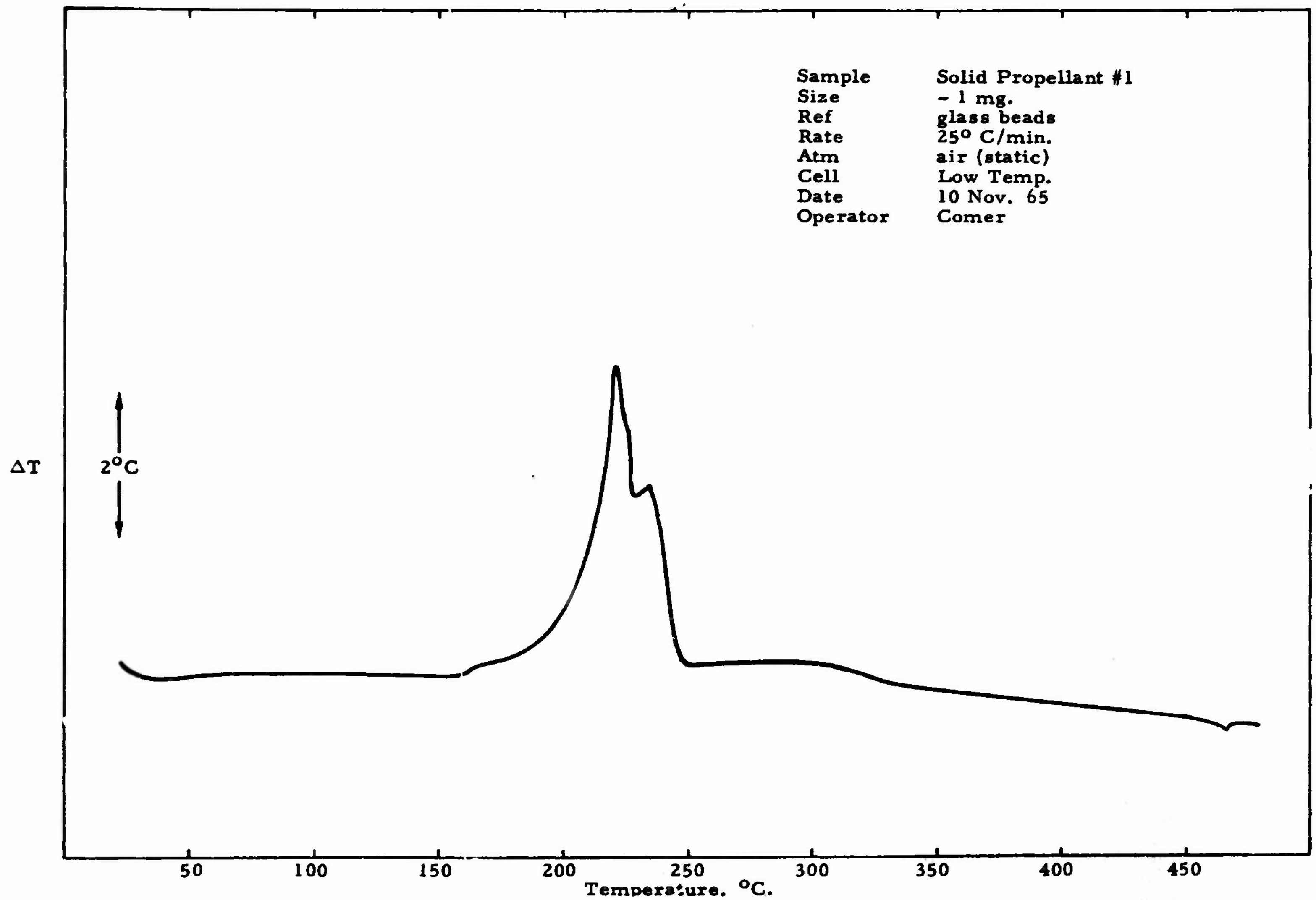

Figure 2. INFO-635 (Washed with Freon-11)

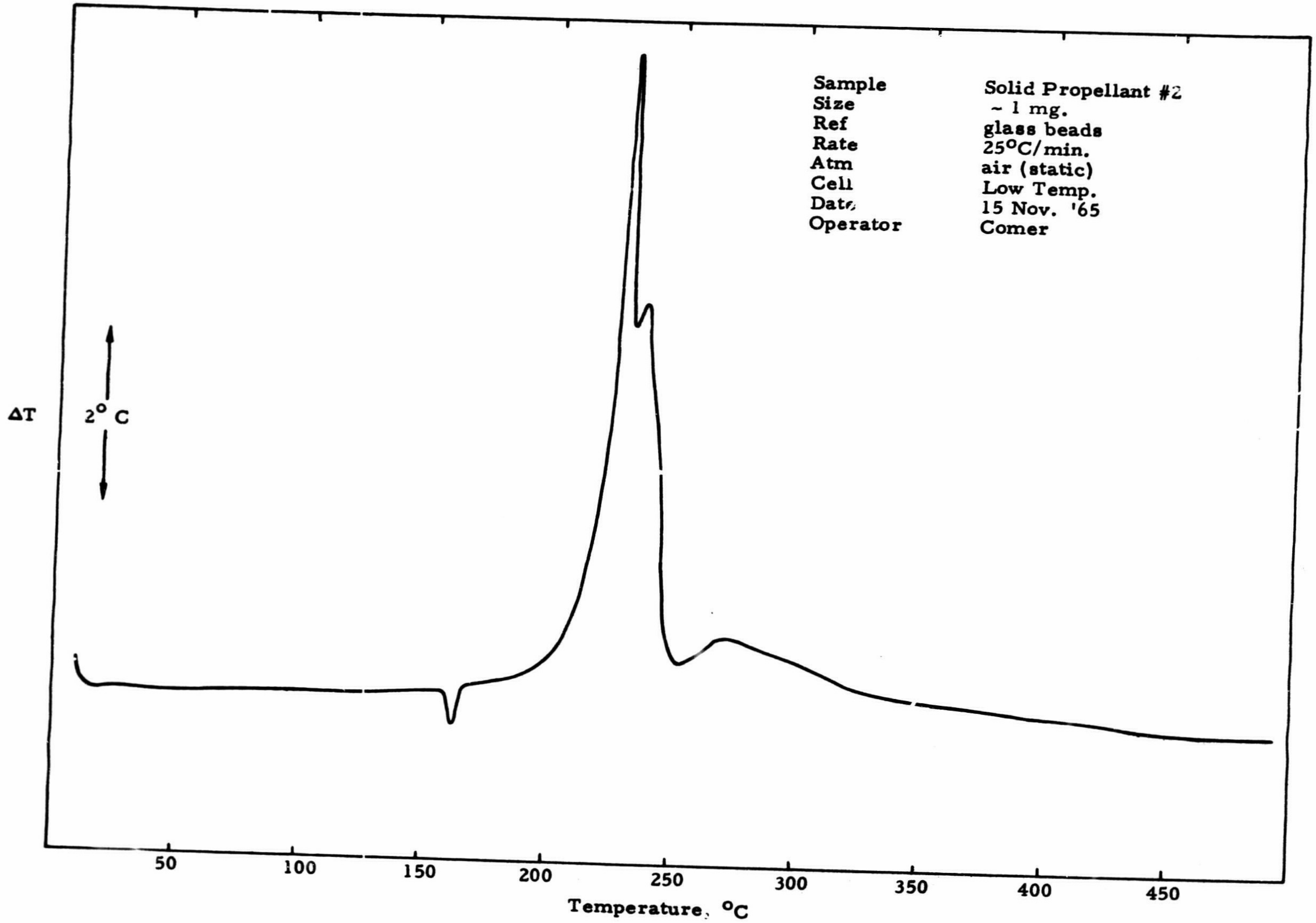

Figure 3. INFO-635 (Water washed)

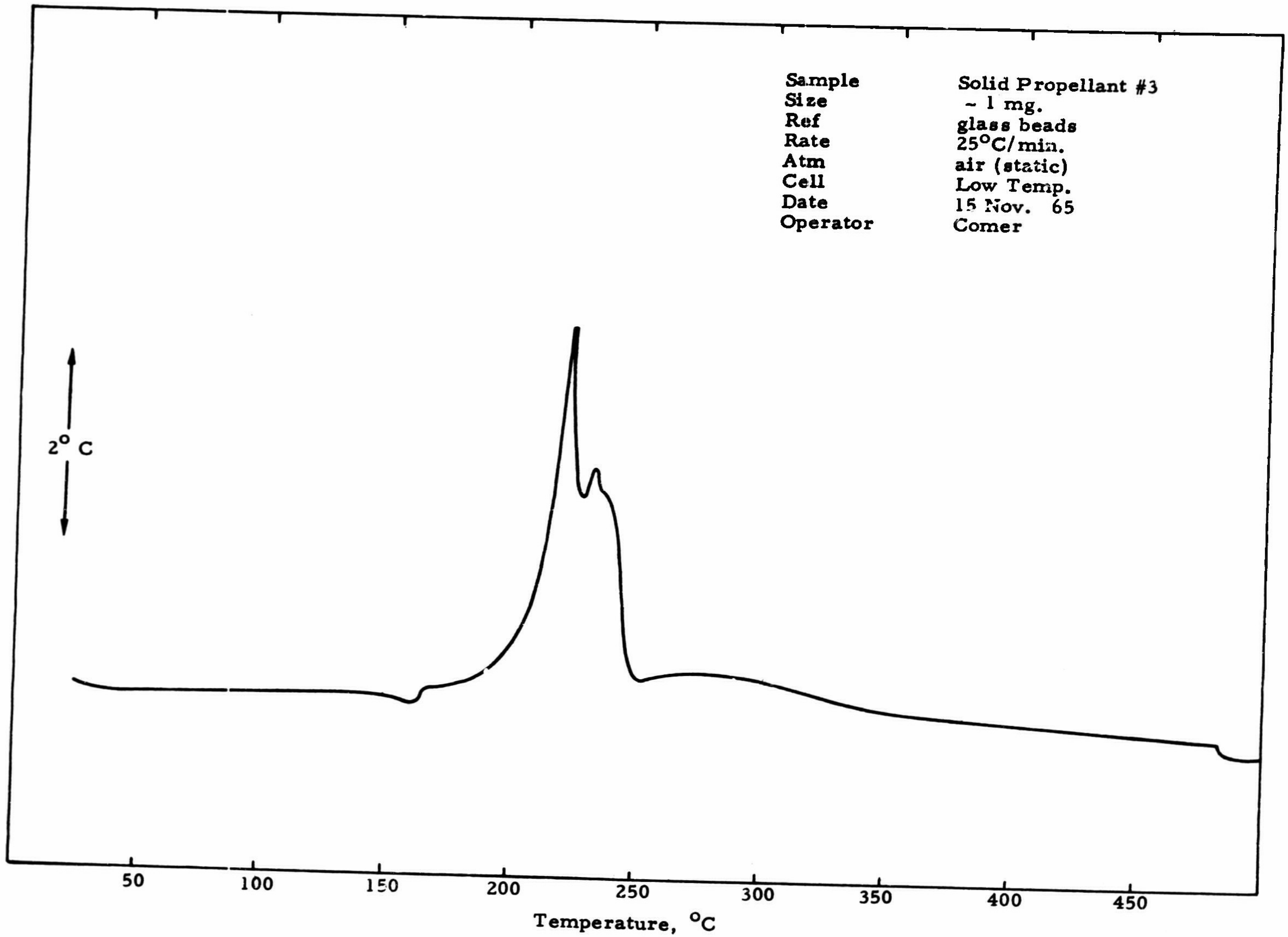

Figure 4. INFO-635 (Washed with Freon-113)

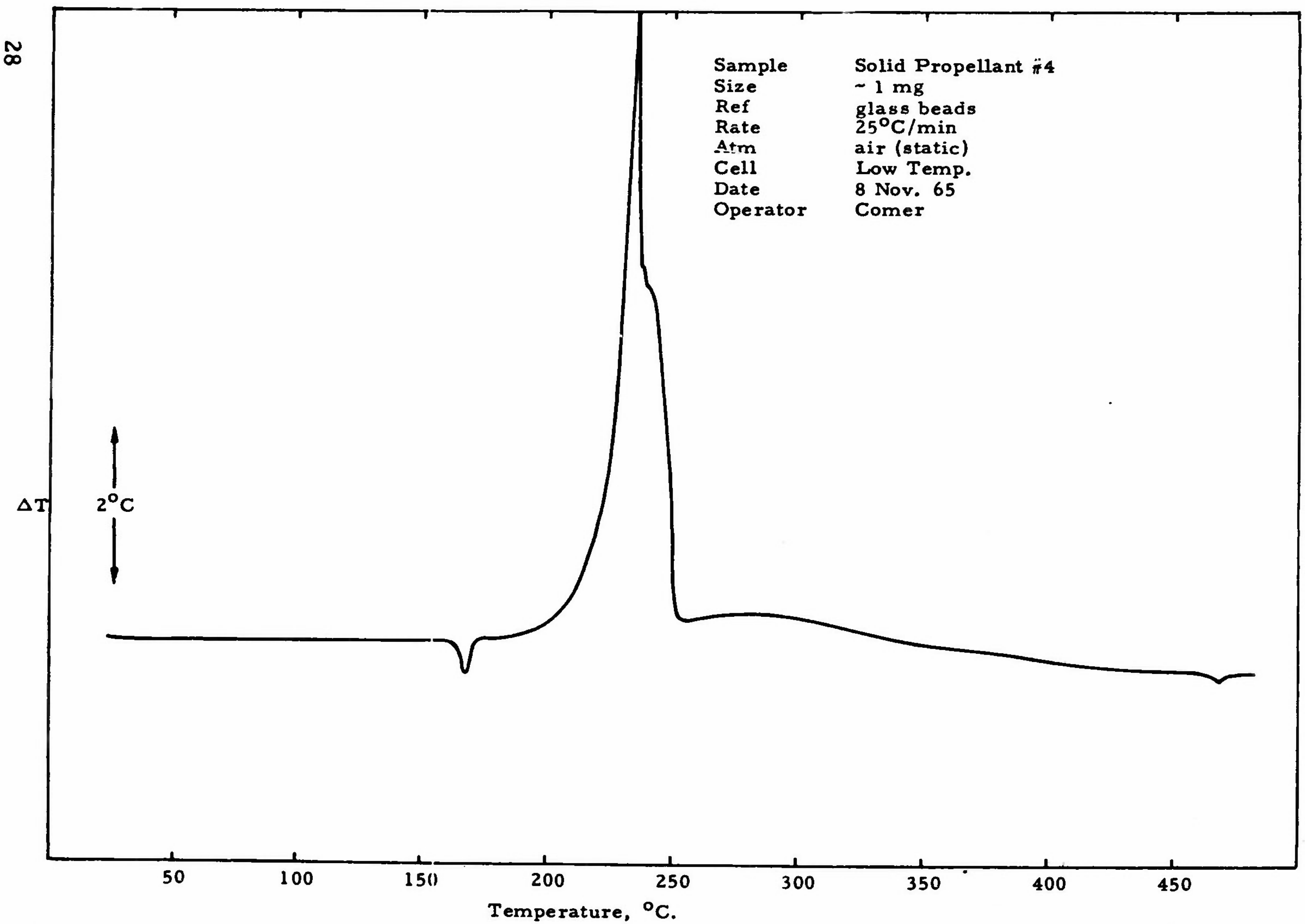

Figure 5. INFO-635 (Once chromatographed)

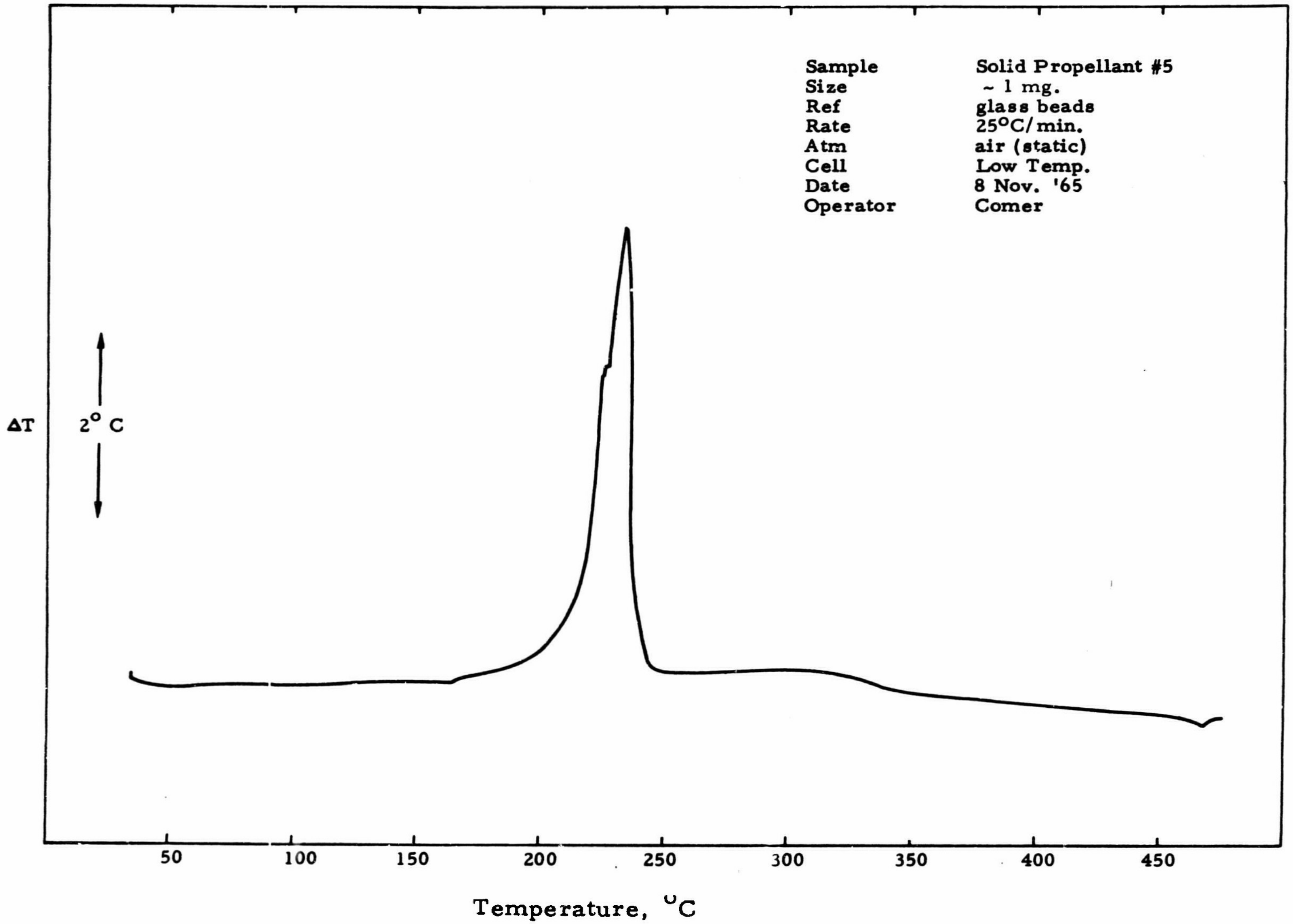

Figure 6. Crude INFO-535 (Tentative) (Washed with Freon-113)

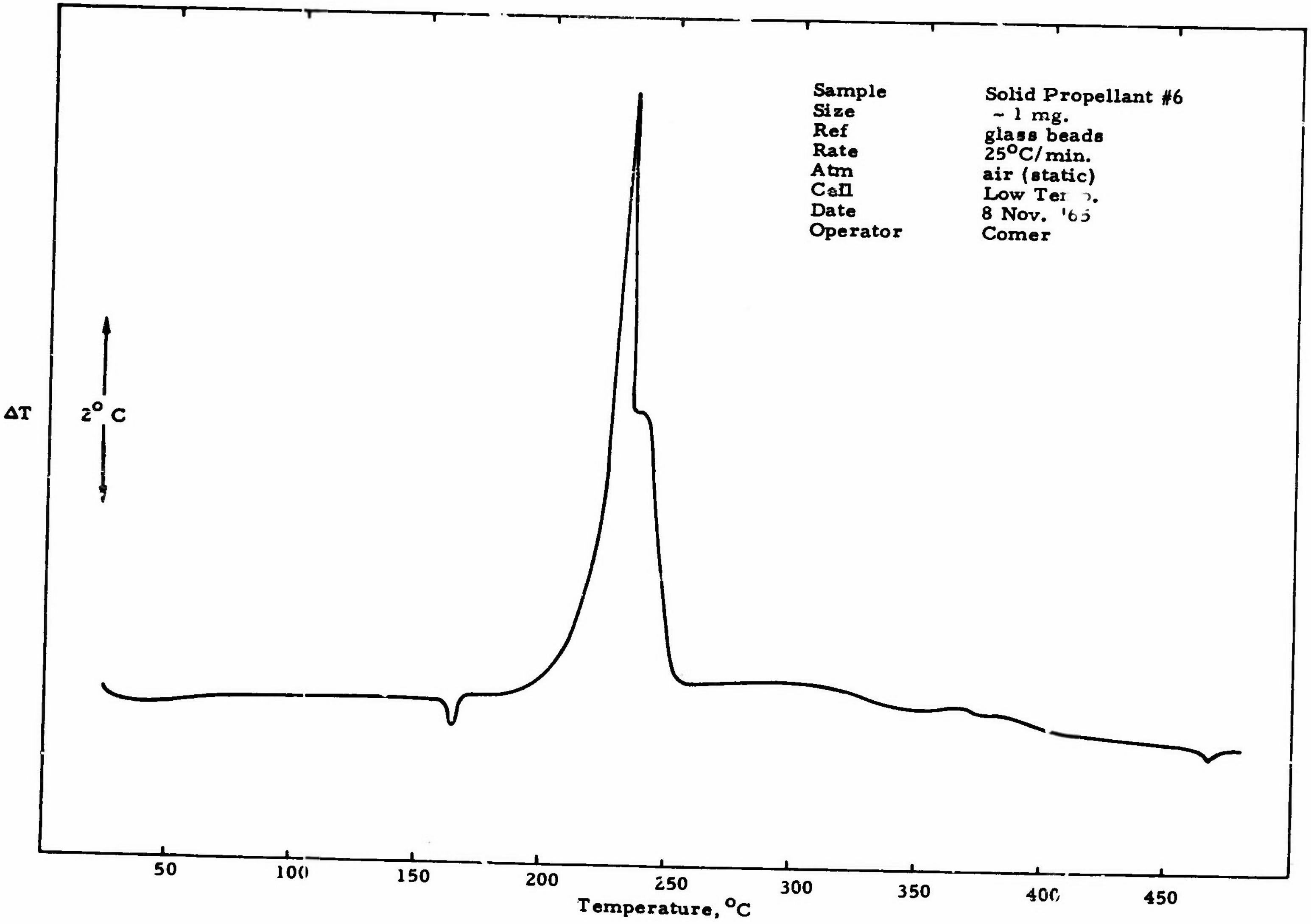

Figure 7. INFO-635 (Twice chromatographed)

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6. Esso Research and Engineering Company, "Desensitization of Available High-Energy NF Compounds," contract No. AF 04(611)-9969, Annual Report, July 1965, (Confidential).

7. Private Communication between Dr. W.J. Leahy, AF Rocket Propulsion Laborator, and Dr. J.A. Brown, Esso Research and Engineering Company.

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Department of the Air Force AFSC, RTD Air Force Rocket Propulsion Lab, Edwards, Calif.	2a. REPORT SECURITY CLASSIFICATION Confidential 2b. GROUP 4

3. REPORT TITLE

Solid Propellant Exploratory Evaluation Semiannual Report No. 3

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Semiannual report July 1965 - December 1965

5. AUTHOR(S) (Last name, first name, initial)

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6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
June 1966	31	10

8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S)
b. PROJECT NO. 314804	AFRPL-TR-66-123
c.	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)
d.	

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13. ABSTRACT

(U) Composite propellants with high solids loadings were made to gain an in-house capability in processing highly viscous composite propellants. Viscosities as high as 70 kilopoise were encountered with solids loadings at 86%. Batch sizes varied from 15 grams to 4 pounds. Burn rate, propellant density, and Shore A hardness were determined for the formulations processed.

(U) Thermal stability determinations were made on three samples of LMH-1 produced by Olin Mathieson during attempts to prepare a more stable material. The best sample underwent 1% decomposition in 660 hours at 60°C. Double-base propellant samples were formulated. The most stable sample yielded the most stable propellant even though the propellant density upon curing was the worst of the three propellant samples.

(U) No improvements in friction sensitivity were observed when samples of INFO-635 were washed with Freon 11 or 113, which is in direct contrast to the improvement in impact sensitivity noted by this laboratory. A solid material (probably Compound 535) has been isolated, and samples of this material have been subjected to impact and friction tests. Differential thermal analysis of a variety of samples of INFO-635 indicated that some ultrasensitive ingredients may have been removed from INFO-635 by the Freon treatments.

DD FORM 1 JAN 64 1473

UNCLASSIFIED

Security Classification

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14. KEY WORDS	LINK A ROLE	LINK A WT	LINK B ROLE	LINK B WT	LINK C ROLE	LINK C WT
Composite Propellant						
LMH-1						
INFO-635						
Solid Propellant Evaluation						

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